

COMPARATIVE STUDY OF THE EFFECT OF IMIDAZOLE AND TRIETHYLENTETRAMINE ON THE CORROSION BEHAVIOR OF API-5L-X65 STEEL IN 1M HCl ENVIRONMENT

SHITTU, S. A^{1*}, OLORUNTOBA, D. T², ARIBO, S³, ADEDIRAN, A. A⁴ & OGUNBADEJO, A. S⁵

^{1,2,3,5}Department of Metallurgical and Materials Engineering, Federal University of Technology Akure, Ondo State, Nigeria

⁴Department of Mechanical Engineering, Landmark University, Omu-Aran, Kwara State, Nigeria

ABSTRACT

This research focused on studying the corrosion behavior of API-5L-X65 steel in acidic environment (1M HCl) containing Triethylenetetramine (TETA) and Imidazole (IMI) corrosion inhibitors. Corrosion rate of the API steel coupons were evaluated in 1M HCl at room temperature using both gravimetric and Potentiodynamics polarization techniques. The corroded steel in the HCl was characterized using optical microscope and atomic absorption spectrometer. The result revealed that with increase in volume of the IMI and TETA compounds, there was a proportionate decrease in the rate at which the API steel corrodes in the acidic environment. Also, corrosion rate of 6.0748E-09 mm/yr and 7.0417E-09 mm/yr were observed for the steel sample in 50 ml IMI and 50 ml TETA, respectively. Maximum corrosion efficiency of 89.28% and 87.58% were obtained for 50 ml IMI and 50 ml TETA, respectively. The morphology of the corroded steel surface revealed that IMI adsorbs more than the TETA inhibitor.

KEYWORDS: Corrosion, Triethylenetetramine, Imidazole, Inhibitors & API-5L-X65 Steel

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1 INTRODUCTION

API- 5L- X65 steel is a high-strength low-alloy (HSLA) steel that is mostly used as a construction material for pipelines, pumps, off-shore rigs, tanks, agitators (Sherif *et al.*, 2013). The low cost of API- 5L -X-65 steel relative to the higher performing steels grade is among the reasons, while it is utilized in several applications. In the industrial sector, a major challenge is usually faced by pipeline steels, where they are affected by corrosion consequent to its exposure to corrosive environment, such as HCl (Junaedi *et al.*, 2013). Industrial processes, such as the use of acids for pickling, cleaning, removal of scales as well as oil well acidizing facilitate corrosion and because of the aggressive nature of this acid, inhibitors are utilized in order to minimize the metals dissolution rate (Firdhouse *et al.*, 2013). Organic inhibitors have been reported to be relatively eco-friendly, non-toxic, readily available and sustainable. Their ability to resist decomposition with time as compared to most of the green inhibitors is among the reason why they are the most effective and efficient types of inhibitors. These inhibitor molecules have heterocyclic compounds with polar functional groups (e.g., Nitrogen, Sulfur, Oxygen and Phosphorus) and/or conjugated double bonds with different aromatic systems. Metals are usually protected by inhibitors when the inhibitors adsorb themselves to the substrate and then form a passive layer, thus protecting the metal (Bothi, 2008). Inorganic inhibitors have been put to use in many industrial processes for corrosion protection, but due to some reasons, such as cost, toxicity, preference is given to organic inhibitors (Abdel-Gaber *et al.*, 2009; Asipita *et al.*, 2014; Abdulrahman *et al.*, 2014). Also, there exists organic green inhibitors (natural products), but

they are yet to be produced at commercial level. The use of plant extract as corrosion inhibitors has been in existence for a long time and several researches have been conducted. Green inhibitors from plant extract have been reported to display high inhibition efficiency level in addition to their low cost and availability (Patel *et al.*, 2013). However, the debate is that the plant kingdom is likely to diminish slowly if plant materials are used as corrosion inhibitors, that is metals will be offered protection at the detriment of the plant kingdom. Furthermore, the problem of using some plant inhibitors is their instability, that is they are readily biodegradable (Rajendran *et al.*, 2005). The use of TMT and IMI as corrosion inhibitors has been widely studied and result has revealed that they offer good protection for steel in water. In this present research, the corrosion behavior of API 5L X65 steel in 1M HCl containing various concentrations of TMT and IMI corrosion inhibitors were studied at room temperature.

2. MATERIALS AND METHODS

2.1 Preparation of Steel Samples

The steel samples were cut into test coupons - dimension of length 10 mm x 10 mm x 0.78 mm using a shear cutter. The dimensions were measured using Digital Vernier Caliper. Each coupon was wet polished on a grinding machine mounted with silicon carbide (SiC) emery paper of grit size between 60 and 1200. This was done in order to obtain a mirror-like surface suitable for gravimetric test and the polarization test. Thereafter, they were rinsed in acetone and air dried. Accurate weights of the samples were taken using chemical weighing balance and recorded. Table 1 displays the chemical composition of the tested steel.

2.2 Preparation of Reagents

Analytical chemicals, such as Triethylenetetramine (TETA), Imidazole (IMI), HCl and Distilled water were obtained. Thereafter, 1M concentration of HCL environments were prepared using the HCl and distilled water. Also, concentrated solutions of 0.05 M TETA and IMI inhibitors each were prepared using the analytical chemicals and distilled water. Various volumes of 10, 20, 30, 40 and 50 ml of the prepared solutions were introduced into different plastic beakers containing 150 ml of the prepared HCl.

2.3 Corrosion Rate determination by Weight Loss Method

API-5L-X65 steel sample was immersed into plastic containing 150 ml of 1M HCl without inhibitor, which serves as the control experiment. The prepared volumes of 10, 20, 30, 40 and 50 ml each for both the TETA and IMI inhibitors were poured into plastic beakers containing immersed steel samples and 1M HCl corrosive medium. The weights of each sample were recorded before immersing them into the environment. Thereafter, corresponding weights of the immersed samples were taken at every three days' interval for three weeks using an electronic weighing balance.

The corrosion rate of the samples were computed using equation (1) (Oloruntoba *et al.*, 2017).

$$CR = \frac{W}{A_{365} T}, \quad (1)$$

where CR is the corrosion rate in mg/mm²/yr, W is weight loss in milligrams, A is the total surface area of exposure in mm², T is the exposure time in days.

2.4 Corrosion Rate Determination by Polarization Study

The steel samples were mounted in polyester resin to give an exposed surface area of 10 mm². The polarization study was

performed using a potent iostat, consisting of three electrode cell setups. The API steel was used as the working electrode, while platinum and silver chloride electrode (Ag/AgCl) were the counter electrode and reference electrodes, respectively. The parameters measured during the study were corrosion potential (E_{corr}), corrosion current (I_{corr}) and Tafel plots. The corrosion current (I_{corr}) values were inputted into equation (2) in order to obtain the corrosion penetration rate (CPR).

$$CPR = \frac{K_1 X I_{corr} X EW}{\rho}, \quad (2)$$

where CPR is the corrosion rate (mm/yr), K_1 is a constant $= 3.27 \times 10^{-3}$ (mmg/ μ Acmyr), Equivalent weight (EW) $= 27.7$, density $\rho = 7.86$ g/cm³.

The inhibitor efficiency was measured by equation (3):

$$I.E\% = \frac{CR_{inh} - CR_{blank}}{CR_{blank}} \times 100, \quad (3)$$

where I. Eis inhibitor efficiency (percentage), CR_{inh} is corrosion rate of metal with inhibitor and CR_{blank} is corrosion rate of metal without inhibitor.

2.5 Atomic Absorption Spectroscopy Analysis

The analytical method used for the analysis of metal concentration was spectrometry and the equipment used is Atomic Absorption Spectrophotometer (AAS) Buck Scientific model 210 VGP using the calibration plot method. The AAS was carried out to determine the concentration of iron (II) ions in ppm that dissolved in 1M HCl after 21 days' immersion time in the presence and absence of different volumes of TETA and IMI.

2.6 Surface Analysis

The surface morphology of the API steels consequent to the reaction of the environment with the steel's surface were analyzed with optical microscopy. The API-5L-X65 steel immersed in 1M HCl for 1,200 seconds from the electrochemical test at normal condition was used for the study. Sample in the absence of inhibitor (considered as control) and those exposed at optimum volumes of the inhibitors were subjected to optical microscopic analysis.

3. RESULTS AND DISCUSSIONS

3.1. Chemical Composition

The elemental composition of the API-5L-X65 steel used for this research is displayed in Table 1.

Table 1: Composition of the API- 5L- X65 Steel used

C	Si	Mn	P	S	Cr	Mo	Ni	Al	Co	
0.12	0.002	0.36	0.014	0.013	0.044	0.0006	0.010	0.038	0.008	
Cu	Sn	As	Zr	Ca	Sb	Se	Te	B	N	Fe
0.041	0.002	0.004	0.001	0.0004	0.023	0.012	0.009	0.0003	0.092	99.4

Table 1 represents the elemental composition of the as received API- 5L- X65 steel used for the research. It is observed that the steel is deficient of sufficient alloy component required to resist corrosion tendencies, especially Chromium and Nickel. The percentage carbon composition is 0.12%, which belongs to the class of low carbon steel and that makes it to be ductile and malleable. However, the amount of alloying elements content is less than 2%, that makes it plain carbon steel. Therefore, it is vulnerable to corrosion in chloride environment (Yarong *et al.*, 2017).

3.2 Corrosion Rate of API- 5L- X65 Steel in 1M HCl with and without TETA and IMI Corrosion Inhibitors

Figures 1 and 2 displays the plot of corrosion rates against exposure time for the API-5L-X65 steel in 1M HCl with and without IMI and TETA inhibitors. The plots were obtained from the weight loss results.

The plot of corrosion rate with time for the API 5L X65 steel immersed in 1M HCl without and with IMI inhibitor for 21 days is displayed in figure 1. It was observed that the corrosion rate increased with increase in the inhibitor's volume from 10 ml to 20 ml. The corrosion rate later decreased, as the inhibitor concentration increased from 30 ml to 50 ml. Steel sample in IMI inhibitor concentration of 50 ml had the least corrosion rate of $1.4021\text{E-}06 \text{ mg/mm}^2/\text{yr}$ as against the corrosion rate of $1.5527\text{E-}06 \text{ mg/mm}^2/\text{yr}$ for the control sample.

Figure 2 displays the variation of corrosion rate with time for the API -5L- X65 steel immersed in 1M HCl without and with TETA inhibitor for 21 days. It was observed that the addition of 10 ml TETA inhibitor led to an increment in the corrosion rate of the API steel. The corrosion rate later decreased as the inhibitor concentration increased from 20 ml to 50 ml. Corrosion rate of $1.4629\text{E-}06 \text{ mg/mm}^2/\text{yr}$ at 21 days immersion period was recorded by 50 ml concentration, and this is the least corrosion rate for TETA.

The high corrosion rate was observed for the steel samples, as the inhibitor concentration increased from 10 ml to 20 ml for IMI and at 10 ml for TETA in Figures 3.1 and 3.2, respectively, which could be as a result of the lower surface coverage of the inhibitors on the steel surface, thereby exposing more of the steel surface to the chloride environment. It may also be that at a lower concentration, rather than the cation of the inhibitor becoming attracted electrostatic ally to the anion of the steel surface, it reacted with the HCl forming complex ions that keeps on robbing the steel of its electron. Also, the reduction in corrosion rate, as observed in the increase in the concentration, amount from 30 ml to 50 ml for IMI and from 20 ml to 50 ml for TETA, may likely be as a consequence of the electron-rich N atoms of -NH_2 group present in both IMI and TETA. In the aqueous acidic solutions, IMI and TETA may occur either as neutral molecules or in the form of protonated molecules (cations). Generally, the surface of steel becomes positively charged in acid solution and as a result hinders the protonated inhibitors from being attracted towards the mild steel surface of positive charge due to the electrostatic repulsion. However, chloride ions which are known to bear smaller degree of hydration could bring in excess negative charges around the interface, thus allowing more of the positively charged inhibitor molecules to be adsorbed. Also, the adsorption of the protonated inhibitors is by electrostatic interactions that occur between the positively charged molecules and the negatively charged metal surface. Thus, resulting in a decrease in corrosion rate, as reported by Odozi *et al.* (2013), Abdallah *et al.* (2012), Mehdi *et al.* (2016) and Mohammad *et al.* (2017).

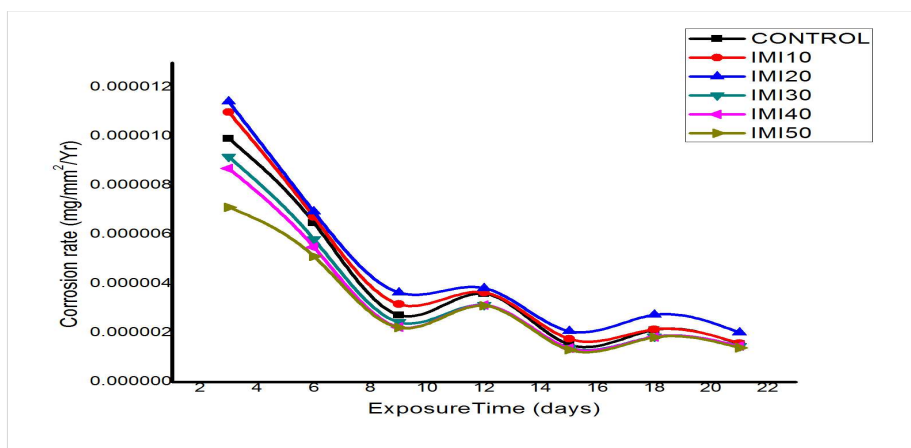


Figure 1: Plot of Corrosion Rate against Exposure Time for the API-5L-X65 Steel Immersed in 1M HCl with and without IMI Inhibitor.

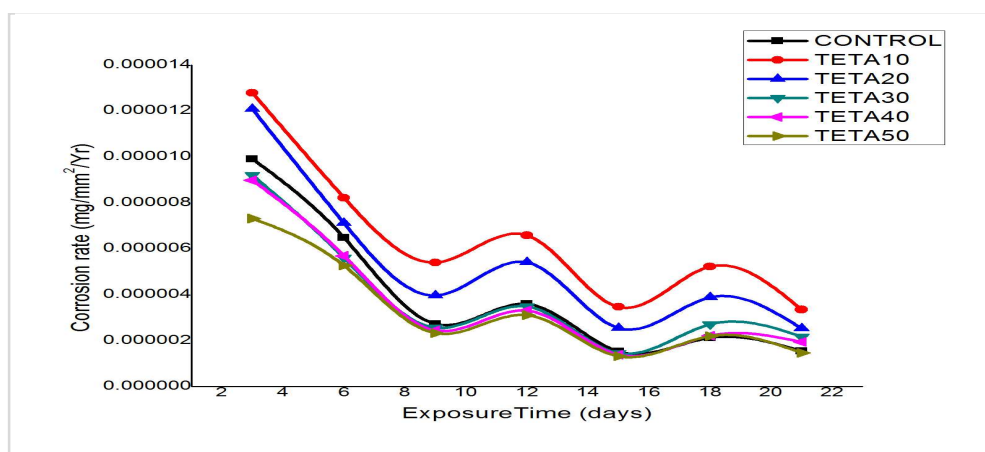


Figure 2: Plot of Corrosion Rate against Exposure Time for the API-5L-X65 Steel Immersed in 1M HCl without and with TETA Inhibitor.

3.3. Atomic Absorption Spectroscopy Results

Figure 3 represents the concentration of dissolved iron present in the selected inhibitor volumes in the 1M HCl environment after 21 days. It was observed that the concentration of dissolved iron drastically reduced with increase in the inhibitor volume. The IMI 50 sample revealed the least concentration of dissolved iron with value 0.796 ppm followed by TETA 50 with value 0.965 ppm. High value of dissolved iron ion implies high corrosion rate and vice versa.

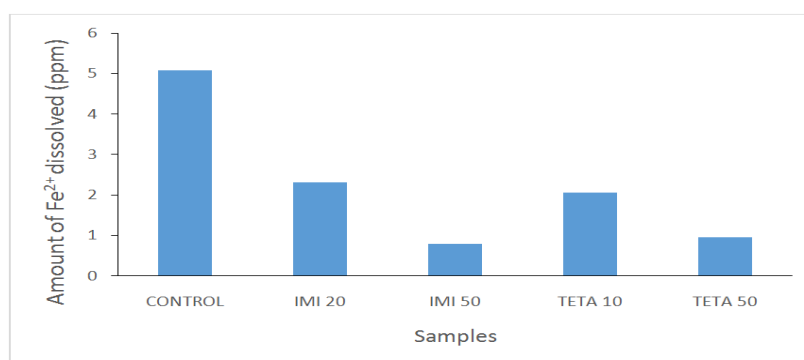


Figure 3: Variation of Dissolved Iron Concentration with Concentration of Inhibitors for the API-5L-X65 Steel in 1M HCl Environment.

3.4. Potentiodynamic Polarization Measurement for API- 5L- X65 Steel in 1M HCl with and without TETA and IM Corrosion Inhibitors

Figure 4 displays the graph of potential versus log of current density for Tafel Polarization of API- 5L- X65 Steel in 1M HCl with and without IMI corrosion inhibitors. Other information is displayed in Table 2. From the curve, a shift of the potential towards the negative direction was observed with the addition of 10 ml IMI, and the current density decreased too relative to the control sample. The potential shift was from -0.4318 V Ag/AgCl to -0.4331 V Ag/AgCl, while the current density was from $4.8834\text{E-}06 \text{ A/cm}^2$ to $3.3192\text{E-}06 \text{ A/cm}^2$, but the potential later shifted to the positive direction, as the inhibitor concentration increased from 20 ml to 50 ml with a potential shift from -0.4309 V Ag/AgCl to -0.4229 V Ag/AgCl and a proportionate decrease in current density from $3.6719\text{E-}06 \text{ A/cm}^2$ to $5.2336\text{E-}07 \text{ A/cm}^2$.

Figure 5 displays the plot of potential versus log of current density for Tafel Polarization of API- 5L- X65 Steel in 1M HCl with and without TETA corrosion inhibitors. More information is displayed in Table 2. From the curve, a proportionate decrease in current density from $4.8834\text{E-}06 \text{ A/cm}^2$ to $6.0667\text{E-}07 \text{ A/cm}^2$, as the increase of inhibitor concentration from 10 ml to 50 ml was observed. The 50 ml concentration had a positive potential shift from -0.4318 V Ag/AgCl to -0.4275 V Ag/AgCl relative to the control.

From the Tafel plots, both of the cathodic and anodic arms for the various inhibitors concentration lies behind that of the blank solution, which indicates that IMI and TETA inhibitors may act as a mixed inhibitor. Also, corrosion current is a kinetic indicator of the corrosion process, a reduction in the corrosion current denotes a lower corrosion rate (high inhibition efficiency). The corrosion current values obtained for the various inhibitors concentration revealed that the API-5L-X65 steel was inhibited in the 1M HCl environment. The corrosion potential recorded for the various inhibitors concentration revealed a close range. The ranges were -0.4331 V to -0.4230 V for IMI and -0.4303 V to -0.4275 V for TETA. The average potential difference for various concentrations is about 0.003 V. In classifying an inhibitor as cathodic or anodic, the difference in their corrosion potential (E_{corr}) should exceed and or be close to 85 mV (0.085 V). Else, it will be considered as a mixed type inhibitor (El-Etre *et al.*, 2007). The values obtained for the corrosion potential of the various inhibitors concentration revealed that the IMI and TETA act as a mixed inhibitor for the API-5L-X65 steel in 1M HCl environment. In addition, a cathodic inhibitor will shift the corrosion potential towards more positive side, while an anodic inhibitor moves it in the negative direction. Mixed inhibitors display the behavior of both anodic and cathodic inhibitors. It can be observed that the E_{corr} shifted to both the positive direction as in the case for the IMI single, TETA single, 20 series, 30 series and 50 series, while that of 10 series and 40 series shifted to the negative direction. This further confirms that the IMI and TETA inhibitors acted as a mixed inhibitor type in the system studied.

Evidently, almost all the IMI and TETA inhibitor concentrations introduced to the 1M HCl environment significantly reduced the rate at which the API-5L-X65 steel corrodes. The 20 ml concentration gave the highest corrosion rate of $4.2621\text{E-}08 \text{ mm/yr}$ for IMI inhibitor, while 50 ml gave the least corrosion rate of $6.0748\text{E-}09 \text{ mm/yr}$. The 20 ml concentration gave the highest corrosion rate of $3.7046\text{E-}08 \text{ mm/yr}$ for TETA inhibitor, while 50 ml gave the least corrosion rate of $7.0417\text{E-}09 \text{ mm/yr}$. The highest corrosion rate observed may be as a result of small amount of surface coverage (θ) of the organic inhibitors on the steel surface, thus exposing more part of the steel to the aggressive chloride environment and vice versa. The corrosion inhibition efficiency can also be seen to increase up to 89.28% and 87.58% with the addition of 50 ml IMI and 50 ml TETA inhibitor, respectively.

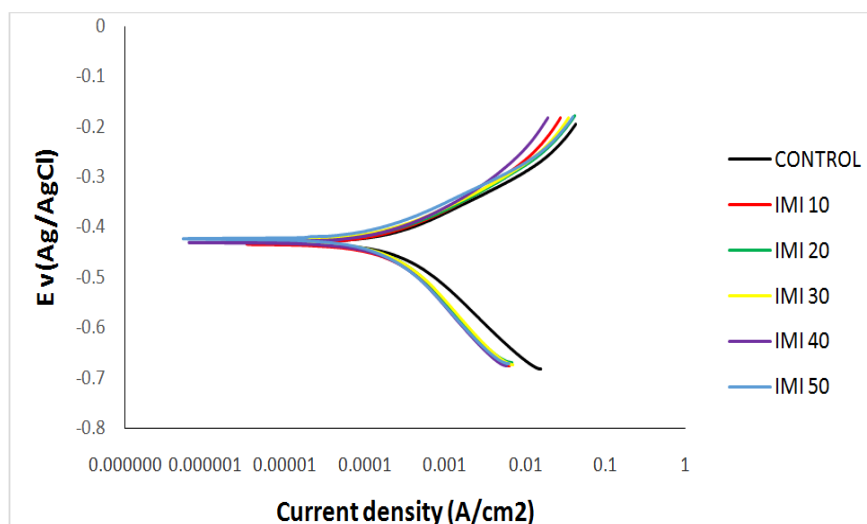


Figure 4: Potentiodynamic Curve for API- 5L- X65 Steel in 1M HCl with and without IMI Corrosion Inhibitors.

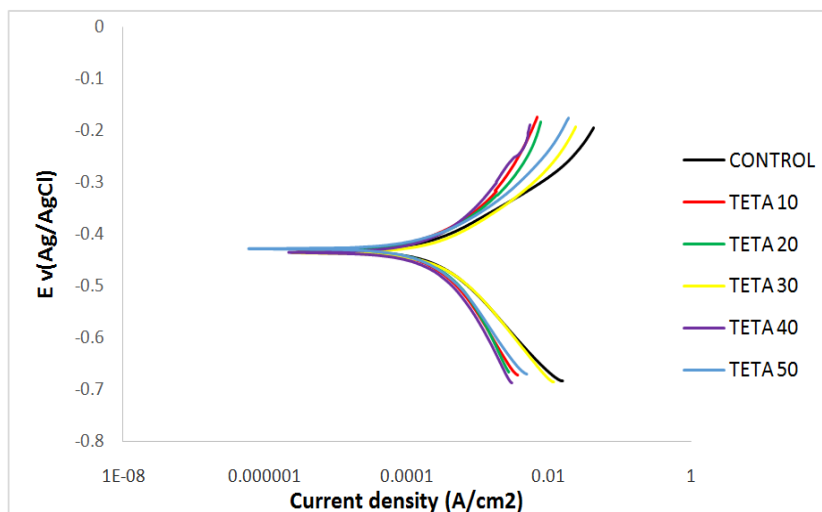


Figure 5: Potentiodynamic Curve for API- 5L- X65 Steel in 1M HCl with and without TETA Corrosion Inhibitors.

Table 2: Data obtained from Potentiodynamic Polarization Measurement

Samples	Corrosion Current Density I_{corr} ($\mu A/cm/Yr$)	Corrosion Potential E_{corr} (mV)	Corrosion Rate (mm/Yr)	Surface Coverage (θ)	Inhibition Efficiency (IE)
CONTROL	4.8834E-06	-0.4318	5.6683E-08		
10 IMI	3.3192E-06	-0.4331	3.8527E-08	0.3203	32.03
20 IMI	3.6719E-06	-0.4309	4.2621E-08	0.2481	24.81
30 IMI	2.5217E-06	-0.4285	2.9270E-08	0.4836	48.36
40 IMI	6.4224E-07	-0.4312	7.4547E-09	0.8685	86.85
50 IMI	5.2336E-07	-0.4229	6.0748E-09	0.8928	89.28
10 TETA	3.0137E-06	-0.4303	3.4981E-08	0.3829	38.29
20 TETA	3.1916E-06	-0.4288	3.7046E-08	0.3464	34.64
30 TETA	2.4021E-06	-0.4358	2.7882E-08	0.5081	50.81
40 TETA	2.1690E-06	-0.4275	2.5176E-08	0.5558	55.58
50 TETA	6.0667E-07	-0.4275	7.0417E-09	0.8758	87.58

3.5 Morphological Studies

Figure 6 (A-D) displays the micrograph of the API-5L-X65 steel before immersion and after immersion in the 1M HCl containing the IMI and TETA corrosion inhibitor. Figure 6A is displaying the morphology of the API-5L-X65 steel sample, which has not been immersed into the corrosive environment. Hence, it is as received without any formation of corrosion product on the surface. It is observed from the micrograph in Figure 6B that there was a formation of a non-tenacious corrosion product on the control sample surface after immersion in the HCl environment. The non-tenacious corrosion product formed as a result of reaction of the steel with the aggressive environment. Thus, increasing the rate at which the steel corrodes. Figure 6C represents the micrograph of the API steel immersed in the HCl environment, containing 50 ml volume of IMI inhibitor. It was observed that the IMI inhibitor adsorbed and was uniformly dispersed on the steel surface, thereby acting as a protective barrier between the steel surface and the corrosive environment. This led to a reduction in the corrosion rate of the steel. Also, Figure 6D represents the surface feature of the API steel after immersion in the HCl environment, containing 50 ml volume of TETA corrosion inhibitor. It was observed that the inhibitor adsorbed uniformly on the steel's surface, thereby offering it more corrosion resistance and to the aggressive chloride environment. However, the API steel in the presence of IMI revealed more surface coverage and least corrosion rate than when it was in the TETA.

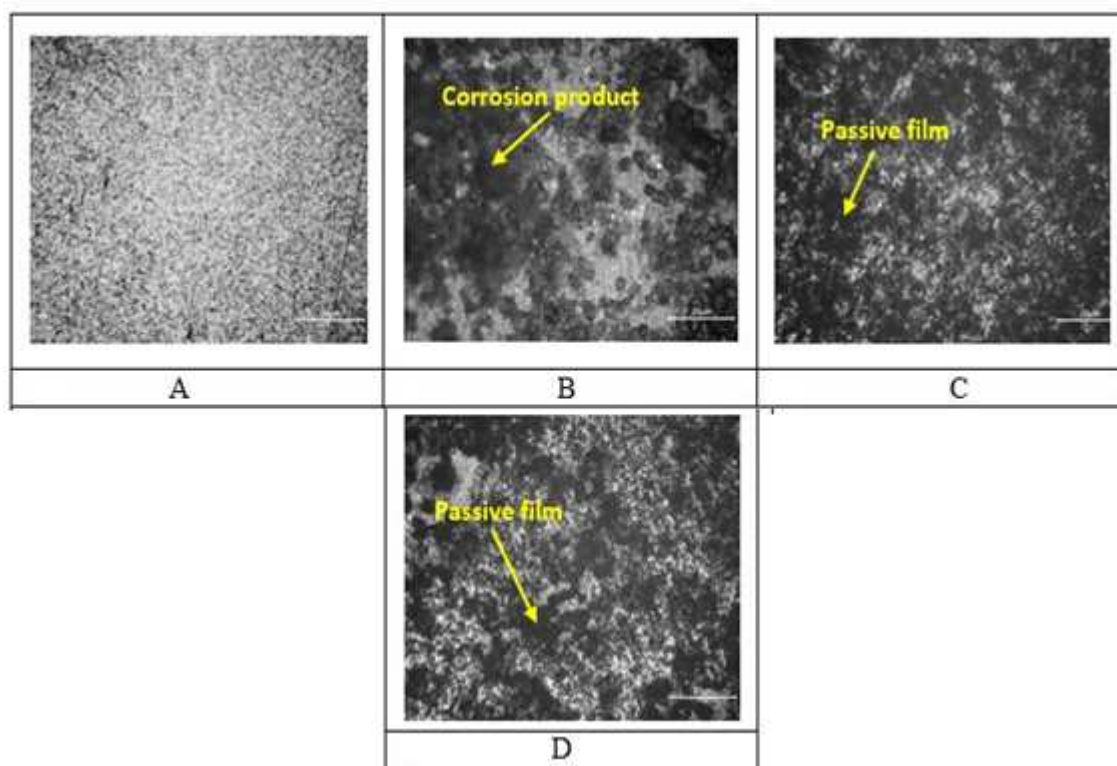


Figure 6: Morphology of API Steel Surface before Immersion (A) and after Immersion in HCl for Control (B) IMA 50 (C) and TETA 50 (D) Samples.

CONCLUSIONS

The results obtained from this study suggest that IMI and TETA are good organic inhibitors for combating industrial corrosion of API 5L X65 steel in chloride environment. Also, the IMI inhibitor revealed the least corrosion rate, high surface coverage and highest inhibition efficiency than the TETA inhibitor.

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AUTHOR'S PROFILE



Shittu Samuel Akinyemi holds a B.Eng., and M. Eng., in Metallurgical and Materials Engineering from the Federal University of Technology Akure. He is currently running his PhD in the same Department. He is a research assistant at the Department of Metallurgical and Materials Engineering, Federal University of Technology Akure. His research areas are in physical and mechanical metallurgy, surface Engineering, corrosion and mineral processing.



Daniel Toyin Oloruntoba is a registered Engineer with the Council for Regulation of Engineering in Nigeria (COREN). He is an Associate Professor in the Department of Metallurgical and Materials Engineering from the Federal University of Technology Akure. A seasoned researcher and member of several professional bodies. His area of research is Corrosion Engineering and Surface Treatment. He has published works on production of anodes for cathodic protection of steel, production of inhibitor for steel, aluminium and copper in corrosive environment. Treatment of metal surfaces by various coating methods. He has supervised undergraduates, post graduates students and attended different conferences.



AdeoluAdesojiAdediran is Lecturer in the Department of Mechanical Engineering, Landmark university, Omu-Aran, Kwara State, Nigeria. He is a registered Engineer with the Council for Regulation of Engineering in Nigeria (COREN) and member of different society. He holds a B.Eng., M.Sc., and PhD in Metallurgical and Materials Engineering. His area of research are in composites development, tribocorrosion studies, conversion coating and mechanical behaviour of materials. He has supervised undergraduate students and attended several conferences.